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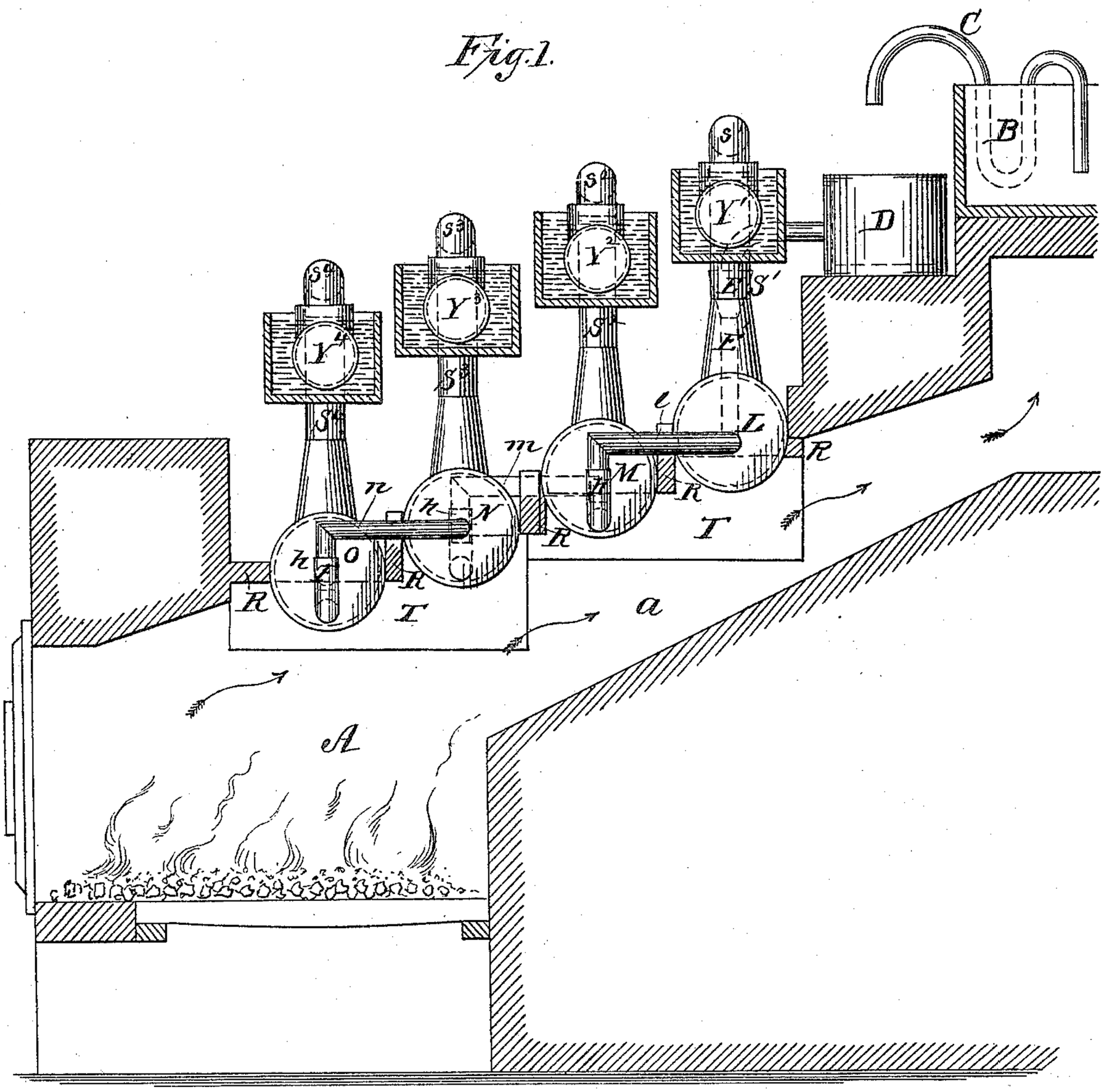
M. WILLETT.

APPARATUS FOR CONCENTRATING SULPHURIC ACID.

No. 301,033. Patented June 24, 1884.

Fig. 1.



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(No Model.) 3 Sheets—Sheet 2.

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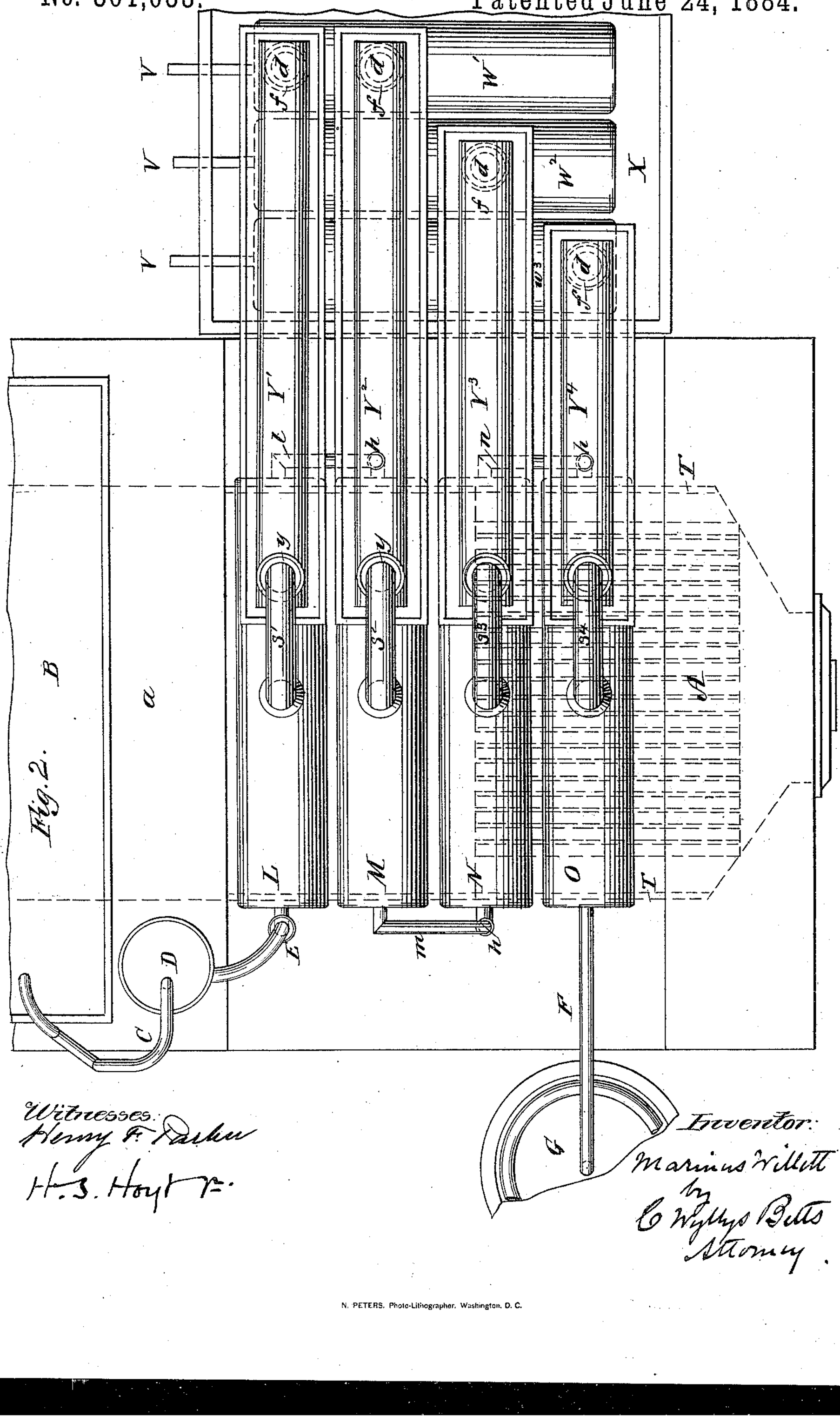

Fig. 2.

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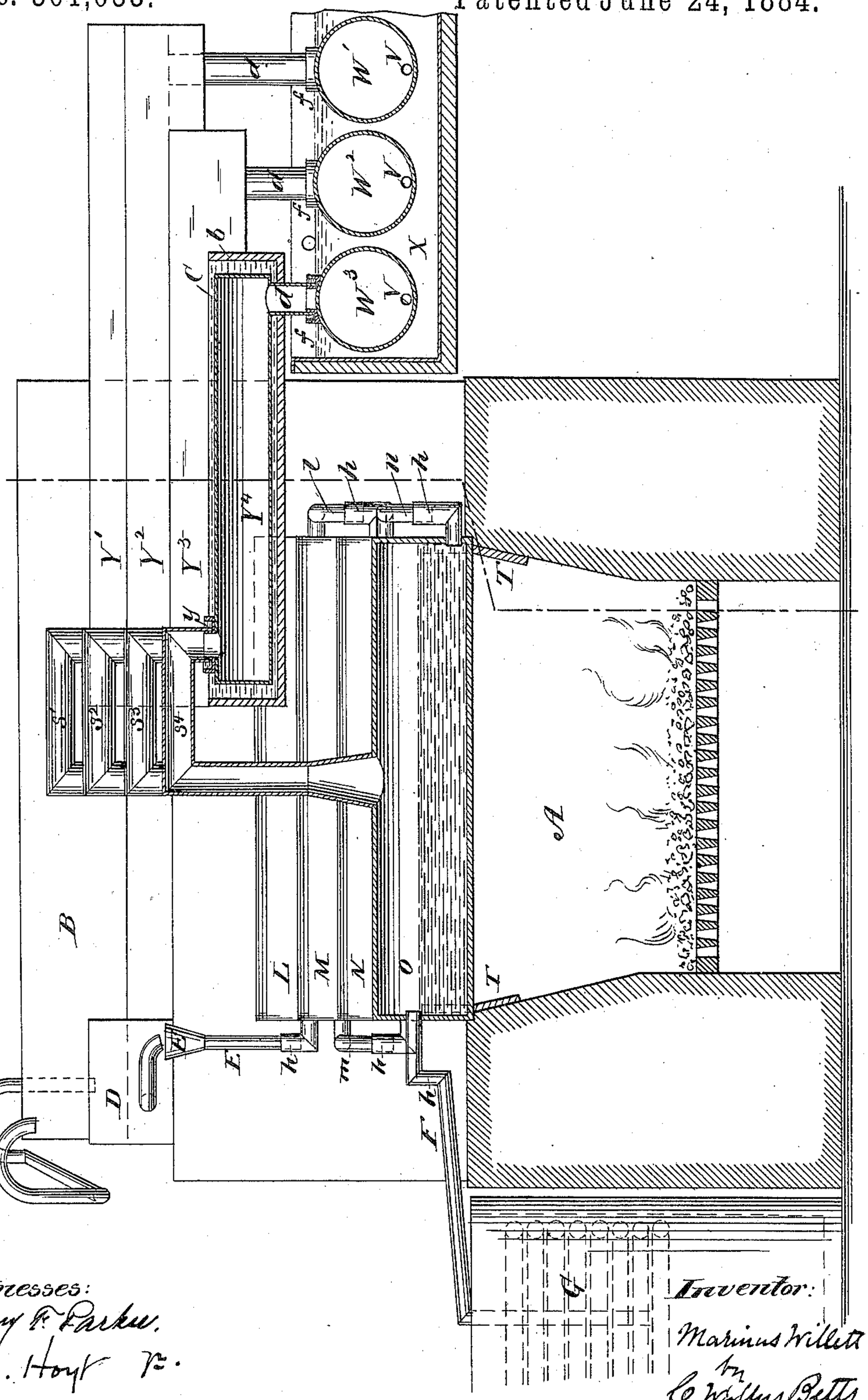

Fig. 3.

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UNITED STATES PATENT OFFICE.

MARINUS WILLETT, OF NEWPORT, RHODE ISLAND.

APPARATUS FOR CONCENTRATING SULPHURIC ACID.

SPECIFICATION forming part of Letters Patent No. 301,033, dated June 24, 1884.

Application filed January 2, 1884. (No model.)

To all whom it may concern:

Be it known that I, MARINUS WILLETT, a citizen of the United States, residing at Newport, in the county of Newport and State of Rhode Island, have invented a new and useful Improvement in Apparatus for Concentrating Sulphuric Acid, of which the following is a specification.

My invention relates to improvements in apparatus for concentrating sulphuric acid to produce oil of vitriol, in which the sulphuric acid is subjected to heat in platinum boilers. Heretofore all such boilers that have been in practical use have been constructed either with a large platinum boiler, or with a platinum dish with leaden water-dome, as shown in the Letters Patent granted to Fauré and Kessler, No. 158,924, dated January 19, 1875, and modifications thereof. The gas produced from the sulphuric acid in the platinum dishes of Fauré and Kessler being condensed under the water-dome above the stills, allowed the upper surface of the acid to be cooled, while the lower surface was heated to the highest possible degree, thus greatly limiting the amount of oil of vitriol produced per square inch of heating-surface. The great diameter of the platinum dishes in all apparatus heretofore used made it necessary to use very heavy platinum, prepared by the expensive process of hammering, in order to support the weight of the acid contained therein, and the large size of these dishes, and the said unavoidable cooling under the water-dome, required a large amount of fuel to heat the acid.

The objects of my invention are, first, by the shape, position, and connections of my boilers, to allow the acid to be more rapidly and easily and more highly heated; second, to condense the gas away from the place where the acid is heated, so that comparatively little cooling action takes place upon the upper surface of the acid; third, to produce more oil of vitriol per square inch of heating-surface; fourth, by the shape and construction of my boilers to allow the use of much lighter platinum, and of rolled instead of hammered platinum, thus greatly reducing the cost; fifth, to reduce the amount of fuel per pound of oil of vitriol produced; sixth, to reduce the size of the apparatus, and thus the cost of the lead and brick plant in the construction of the apparatus; seventh, to reduce the loss of platinum through action of the heat and acid. I attain these objects by the apparatus illustrated in the accompanying drawings, in which—

Figure 1 is a side view of the improved apparatus. Fig. 2 is a view of the same from above. Fig. 3 is an end view of the apparatus, showing one of the improved boilers with its connections.

Similar letters refer to identical parts throughout the several views.

A is the furnace, constructed as usual in apparatus for this purpose, although it may be of much smaller dimensions than heretofore. The heat from the same passes through the inclined shaft *a* and wraps the lower surfaces of the platinum boiler or boilers, and thence passes under the leaden pan B, so as to heat the acid up to a certain point before it passes into the platinum stills. This leaden pan B, containing the supply of sulphuric acid, is constructed in the usual manner, and from it the acid is drawn, by means of the regulating-siphon C, into the leaden boot D, from which it flows into the platinum funnel E, and thence through the supply-pipe E′ into L, which is the first of the series of platinum boilers.

L M N O are the boilers, formed and connected and supported in the peculiar manner, by which I attain the advantages of small cost of construction combined with great producing power. They consist of a horizontal platinum tube, or, preferably, a series of horizontal platinum tubes, at the top of the furnace A and shaft *a*, and arranged, preferably, in descending order toward the furnace A, so that the acid will flow from each into the one next below it by means of the platinum overflow-pipes *l m n*, which are preferably placed at alternate ends of the platinum tubes or boilers, said pipes *l m n* being preferably so arranged that the surface acid of an upper or preceding tube will be carried to the bottom of the tube next below it, as shown in Fig. 1. This method of connection has been found by experiment to create more rapid evaporation of gases than any other, and consequently it makes the apparatus more productive. The depth of acid in the first or highest of the tubes or boilers

should be less than in the next succeeding, and the depth should become greater in each in turn, for the reason that the acid being weaker in the first will reach boiling-point at a lower degree of temperature, and therefore less acid should be allowed to remain in the tube. This construction will prevent the ebullition from overflowing the pipe S for the escape of gases, hereinafter described. I have found that the bottom of the escape-pipe *l*, leading from the first tube or boiler, L, should be preferably about an inch and a half from the bottom of the tube or boiler, and that the bottom of the discharge-pipe F, leading from the last of the series, should be preferably about two and a half inches from the bottom of the last tube or boiler, and that the connecting-pipe, acting as the discharge from each succeeding tube, should be preferably higher than in the one preceding by a fraction of an inch corresponding with the number of tubes in use. This allows a greater depth of acid in the tubes containing the stronger acid and subjected to the most heat. The boiler containing the weakest acid will thus have a film of acid only one and a half inch deep, and the boiler containing the strongest acid will contain a film of acid only two and a half inches deep. The connecting-pipes *l m n* should be made in sections, so that any one tube or boiler can be easily removed and another substituted if repairs are necessary. The joints are indicated at *h*. This is one of the chief advantages of the descending series of tubes, for when arranged thus the connecting-tubes *l m n* may each contain a nicely-constructed slip-joint, *h*, which would not be possible without leakage if the tubes were on the same level. The size and form of these platinum tubes or boilers that I have found useful is that of a cylinder six inches in diameter and three feet long, though I do not confine myself to these dimensions, nor to this shape. The cross-section may be oval or of any other convenient form. In the construction of tubes of this size rolled platinum may be used, instead of the much heavier beaten platinum necessary for the dishes heretofore employed. The reduction in the amount of this metal decreases by many thousand dollars the cost of constructing apparatus yielding a given quantity of oil of vitriol, and my apparatus being much more compact, there is also a great saving of brick-work in building. The producing capacity of my apparatus is much greater than that of Fauré and Kessler, the latter producing but eleven pounds of oil of vitriol per square inch of heating-surface, whereas mine will produce from eighteen to twenty-two pounds per square inch. By the use of these horizontal tubular boilers I also make a great saving by reducing the loss of platinum caused by exposure to heat upon one side and to acid upon the other. Some loss is inevitable, and in the old stills the loss of platinum per ton of oil of vitriol produced was two grams, and in Fauré and Kessler's apparatus the loss was about three one-hundredths (.03) of a gram per ton, while in mine, on account of the small amount of platinum exposed in proportion to the production, the loss of platinum per ton of oil of vitriol produced will be greatly reduced below that of Fauré and Kessler—namely, according to my estimations, to about six ten-thousandths (.0006) of a gram.

I find that the tubes L M N O work well when arranged on the same level, and they may be arranged lengthwise with the shaft *a*, or at any intermediate angle of inclination, provided that each is substantially horizontal. I prefer, however, the descending series shown in the drawings, in order that the difference of acid-level may be maintained, and that the tubes may be made removable by the slip-joints *h*. The series of tubes may be extended to any desired length, the number being limited only by the size of the fire-space in the furnace and the amount of production. A single tube constructed as shown and described, and especially if arranged lengthwise with the flue of the furnace, would be far more productive and economical than any of the apparatus heretofore used; but it would be far less productive than the series of tubes shown, for of course the greater the number of tubes or the greater the length of the tubes the greater will be the amount of acid flowing through the tubes and the amount of production of oil of vitriol; hence, with an increased number of tubes the size of the pipes *l m n* connecting them must be increased. I find that with a series of four tubes, as shown, the best size of connecting-pipe is about one inch, inside measurement, and that if six tubes are used an inch and a quarter pipe will be required. From the last of the series, O, the acid passes, by means of the platinum pipe F, into the cooler G, which is constructed in any ordinary manner, preferably with a coil of leaden pipe, through which cold water passes.

The platinum boilers or tubes L M N O have gas-openings at their tops, preferably in the center, through which the gas produced in each passes, through the platinum pipes $S' S^2 S^3 S^4$, respectively, to the leaden water-arms $Y' Y^2 Y^3 Y^4$, respectively, in which and in the leaden condensers $W' W^2 W^3$, attached, it is condensed in the usual manner. I prefer to have these pipes S about two inches in diameter, and with a flaring mouth about three inches in diameter at the junction with the platinum tube or boiler.

Very weak sulphuric-acid gas being produced from the boilers containing the weak acid, it is of little value, and hence for economy of space the first two water-ways, $Y' Y^2$, and perhaps more in a long series of boilers, may be connected with one condenser, W', as shown in Fig. 3; but the pipes S^4, S^3, &c., leading from the boilers O and N, forming the latter or lower half of the series, and contain-

ing strong acid and giving off strong sulphuric-acid gas, should never be connected with any of the pipes of the earlier or upper half of the series, and the pipe S^4 of the last or lowest boiler, O, must never be connected with any other pipe, for the reason that the tests are made upon the acid condensed from the gas produced by this boiler for regulating the flow of acid into the apparatus from the regulating-siphon C. The pipes S may be of glass, connected with the boilers and condensers by hydraulic joints, but platinum is much better.

The construction of one of the water-arms, Y, is shown in detail in Fig. 3, *b* being the outer casing of wood, without top, containing the water, and *c* being the inner closed condenser of lead surrounded by the water. The pipes S are connected with the leaden casings *c* of each water-way Y by a hydraulic joint, *y*, projecting above the water, as shown in Fig. 3, and from the other end of the casing *c* projects the leaden pipe *d* to the condenser W, this also being connected by a hydraulic joint, *f*. The condensers W′ W^2 W^3 may all be in one water-box, X, as shown in Fig. 3. Both the water-arms Y and the water-box X receive a constant supply of cold water. At the farther extremity of each condenser W are two openings, one at the top (not shown) for the exit of gases that have not been condensed by the cooling process, and another at the bottom, represented by the pipe V, through which passes to a proper receptacle the sulphuric acid produced by the condensation.

The platinum tubes L M N O are supported above the furnace A and shaft *a* by vertical iron plates T, or of other suitable material, Fig. 1, forming the upper portion of the sides of the furnace and shaft. Said plates should be formed to fit the shape of the tubes. Thus the ends of the cylindrical tubes forming the boilers project beyond the direct heat of the furnace, as shown in Figs. 1 and 3, and hence the fine gold solder with which the ends are united to the cylindrical sides of the tubes, as well as the fine gold solder with which the connecting-pipes *l m n* are united to them, will be in no danger of fusion from the heat, these parts being all outside the flue. The tubes or boilers above the acid-level are protected from the flames by pieces of cast-iron, (or other suitable material, R R, Fig. 1,) filling the space between adjoining tubes and extending from the level of the bottom of the overflow-pipe in the upper tube to the level of the bottom of the overflow-pipe in the lower tube. This construction allows the flames to strike the entire surface of the platinum up to the acid-level; but prevents them from striking the tubes above the acid-level. Hence the greatest amount of boiling-surface is obtained for the acid, and no heating-surface whatever for the gas above it. This construction greatly increases the producing power of the apparatus.

I am aware that prior to my invention apparatus for concentrating sulphuric acid have been made with a series of platinum retorts arranged in a plane and connected together by platinum—so-called "siphon"—pipes, which extend from the bottom of one retort to a higher point of the next adjoining retort, said retorts being platinum dishes set into the top of the furnace, in the same manner as vessels are set in ordinary cooking-stoves, and they are covered with conical platinum tops, as shown in the Letters Patent granted to Kolbe and Lindfors, August 30, 1881, No. 246,396. The apparatus shown in this patent is designed to accomplish one of the results secured by my invention—namely, lessening the cost of the apparatus; but it does not greatly, if at all, reduce the cost, for the reason that the forms of the platinum boilers shown would make them very expensive, as they must be constructed of beaten platinum, and probably a larger amount of platinum would be required to construct such apparatus than to construct one of the same producing capacity under the Fauré and Kessler patent, because in the Kolbe and Lindfors patent a very small heating-surface is exposed, and considerable depth of acid exists above the heating-surface, owing to the improper form and position of the connections of the retorts. Hence, for the amount of platinum used there would be a very small production as compared with that of my improved apparatus. I do not regard the Patent No. 246,396 as a practical improvement over the Fauré and Kessler patent, or as practically affecting any of the results of my invention. In view of the series of retorts shown in this patent, however, I do not claim, broadly, the use of a series of platinum retorts connected together; but

What I claim, and desire to secure by Letters Patent, is—

1. In apparatus for concentrating sulphuric acid, the tube-retort L, in combination with the gas-escape pipe S′, the supply-pipe E′, and discharge-pipe *l*, substantially as described.

2. In apparatus for concentrating sulphuric acid, the descending series of two or more platinum boilers connected together by platinum pipes, each having the slip-joint *h*, substantially as described.

3. In an apparatus for concentrating sulphuric acid, a group of boilers arranged over a furnace in a descending series, in combination with a series of plates, as R, between the boilers, having the lower portion of one plate in contact with the lowest boiler of a pair of the series at the level of the liquid, and its highest part in contact with the highest boiler of the two at the level of the liquid, substantially as described.

4. In apparatus for concentrating sulphuric acid, the connected series of two or more platinum tubes, L M N O, supported and protected at the top of the furnace A by the plates T and R, substantially as described.

5. In apparatus for concentrating sulphuric

acid, the descending series of two or more tubular boilers, L M N O, having the acid-level higher in each lower boiler of the series than in the one preceding by means of the relative position of the connecting-pipes and discharge-pipe F, substantially as described.

6. In an apparatus for concentrating sulphuric acid, in combination with a descending series of horizontal tubular boilers, as L M N O, connected substantially as described, whereby a fixed fluid-level is maintained in each, a plate, as R, arranged between each pair of boilers in the series, the lower end of said plate being joined to the lower boiler at its fluid-level, while its upper end is joined to the upper boiler at its fluid-level, substantially as and for the purposes specified.

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